PB90-245622

Liquid Chromatographic Determination of 5-(Methylamino)-2-Phenyl-4-(3-(Trifluoromethyl)phenyl)-3-(2H)-Furanone in Soil

(U.S.) Environmental Research Lab., Athens, GA

1990

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1. REPORT NO.	2.	3. PB90-245622
EPA/600/J-90/062		

4. TITLE AND SUBTITLE
LIQUID CHROMATOGRAPHIC DETERMINATION OF 5-(METHYLAMINO)-2-PHENYL-4-[3-(TRIFLUROMETHYL)PHENYL]-3-(2H)-FURANONE IN SOIL

5. REPORT DATE

6. PERFORMING ORGANIZATION CODE

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8. PERFORMING ORGANIZATION REPORT NO.

9. PERFORMING ORGANIZATION NAME AND ADDRESS
*University of Georgia, Athens GA 30605
**U.S. Environmental Protection Agency, Athens GA 30613

10. PROGRAM ELEMENT NO.
CCUL1A

11. CONTRACT/GRANT NO.

12. SPONSORING AGENCY NAME AND ADDRESS
Environmental Research Laboratory - Athens, GA
Office of Research and Development
U.S. Environmental Protection Agency
Athens GA 30613

13. TYPE OF REPORT AND PERIOD COVERED

14. SPONSORING AGENCY CODE
EPA/600/01

15. SUPPLEMENTARY NOTES
Journal of the Association of Official Analytical Chemists 73(2):298-299, Feb. 1990

16. ABSTRACT

A rapid, sensitive method is described for the determination of 5-(methylamino)-2-phenyl-4-[3-(trifluromethyl)phenyl]-3-(2H)-furanone (RE-40885) concentrations in three soil types. The method consists of extraction of soil samples with methanol, filtration, liquid chromatographic separation of methanol-soluble components using a C_{18} column and UV detection at 275 nm. Recoveries were 94, 96, and 94% from the Greenville, Cecil, and Dothan soils, respectively. Average relative standard deviation was 8.0% in the Greenville soil. The qualitative limit of detection was 20 ppb and the limit of quantitation was 40 ppb in 25-g soil samples.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	3
	20. SECURITY CLASS *(This page)* UNCLASSIFIED	22. PRICE A01

EPA Form 2220-1 (Rev. 4-77) PR

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Reprinted from the *Journal of the Association of Official Analytical Chemists*
Vol. 73, No. 2, 1990

PB90-245622

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EPA/600/J-90/062

Liquid Chromatographic Determination of 5-(Methylamino)-2-Phenyl-4-[3-(Trifluoromethyl)phenyl]-3-(2*H*)-Furanone in Soil

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A rapid, sensitive method is described for the determination of 5-(methylamino)-2-phenyl-4-[3-(trifluoromethyl)phenyl]-3-(2*H*)-furanone (RE-40885) concentrations in 3 soil types. The method consists of extraction of soil samples with methanol, filtration, liquid chromatographic separation of methanol-soluble components using a C_{18} column, and UV detection at 275 nm. Recoveries were 94, 96, and 94% from the Greenville, Cecil, and Dothan soils, respectively. Average relative standard deviation was 8.0% in the Greenville soil. The qualitative limit of detection was 20 ppb and the limit of quantitation was 40 ppb in 25 g soil samples.

A new herbicide, 5-(methylamino)-2-phenyl-4-[3-(trifluoromethyl)phenyl]-3-(2*H*)-furanone (RE-40885), synthesized by Chevron Chemical Co. (Richmond, CA, USA), is being investigated for use in peanuts, cotton, cereals, and grain sorghum (1, 2). Probable field application rates are 0.56–0.84 kg/ha (unpublished data). Due to the newness of the herbicide, methods for its analysis in soil samples have not been described. In this report, methodology developed for the determination of RE-40885 at ppb to ppm levels in 3 soil types using an external standard liquid chromatographic method with UV detection will be described.

Experimental

Apparatus and Reagents

(a) *Liquid chromatograph.*—Model SCL-6A control unit, SIL-6A auto-injector, LC-6A solvent delivery system, and SPD-6A variable wavelength detector set at 275 nm (all from Shimadzu, Kyoto, Japan). Model 3390A reporting integrator (Hewlett-Packard Co., Analytical Group, Palo Alto, CA 94303).

(b) *Analytical column.*—25 cm × 4.6 mm id 5 μm LC-C_{18}, in-line 1 cm × 1.5 mm pellicular C_{18} guard column (Alltech, Chicago, IL, USA).

(c) *Solvents.*—LC grade (J. T. Baker Inc., Phillipsburg, NJ 08865).

(d) *Mobile phase.*—Methanol–water (72 + 28 v/v) at flow rate of 1.0 mL/min.

(e) *Analytical standards.*—RE-40885 (Chevron, Richmond, CA, USA); atrazine (Ciba-Geigy, Raleigh, NC, USA); benefin and trifluralin (Elanco, Greenfield, IN, USA); alachlor (Monsanto, St. Louis, MO, USA); and chlorothalonil (Fermenta, Mentor, OH, USA).

Soil Selection

Soil was collected from 3 areas to represent major soil associations in the southeastern United States. Soil 1 is a Greenville sandy clay loam, soil 2 is a Cecil loam (typical of the eroded piedmont area), and soil 3 is a Dothan loamy sand (typical of large areas of the coastal plain) (Table 1). Soil samples were air-dried and passed through a 10 mesh (2 mm) sieve before analysis. All samples were collected from areas with no known previous exposure to RE-40885.

Extraction

The UV spectra of RE-40885 was obtained using a scanning spectrophotometer (Perkin-Elmer 559A, Oak Brook, IL). The purity of the RE-40885 analytical material was established using gas chromatography/mass spectrometry (GC/MS) (Hewlett-Packard 5840-5985B). Various combinations of acetonitrile–water, methanol–water, and acetonitrile–2-propanol were evaluated for herbicide retention time and capacity factor (k′) (3). The accuracy of the analysis was examined by injection of a series of standards containing 5–700 ng/mL RE-40885 in methanol. This range corresponded to herbicide soil concentrations of 10–1400 ppb, using the described methodology.

Analytical herbicide (in methanol) was added to triplicate flasks containing 25 g of soil 1 to achieve RE-40885 concentrations of 40, 50, 80, 100, 120, 160, 200, 250, 500, and 1000 ng/g soil. After addition of the herbicide, the methanol was removed under a gentle air stream. The soil/herbicide mixture was mixed thoroughly with a wrist-action shaker for 60 min and allowed to equilibrate statically for 24 h. The soil was extracted with 50 mL methanol by shaking for 60 min on a wrist-action shaker. The soil extracts were then filtered through 2 Whatman No. 1 filter papers (Whatman, Clinton, NJ, USA) to remove particulates, and 50 μL aliquots were injected and compared to RE-40885 standards. Soils 2 and 3 were similarly spiked with 200 and 1000 ng/g soil.

Twenty-five g of each soil was spiked with RE-40885 (0.4 μg/g), atrazine (3.57 μg/g), benefin (2.20 μg/g), trifluralin (1.90 μg/g), alachlor (3.10 μg/g), and chlorothalonil (1.10 μg/g). Triplicate flasks for each soil were analyzed with the described method.

Results and Discussion

The UV spectra of RE-40885 showed an absorbance maximum at 275 nm. GC/MS analysis of RE-40885 (analytical grade) indicated a purity of > 99%. Injection of 50 μL of RE-40885 standard produced a peak with a retention time of 2.7–22.6 min, depending on the mobile phase (Table 2). On the basis of k′ values, we selected a mixture of methanol–water (72 + 28) which resulted in a retention time of approximately 7 min (Figure 1a). A standard curve plot was linear in the 10–1400 ppb range (soil basis), with an $r^2 = 0.99$ (data not shown.) The analytical standards (in methanol) and the methanol soil extracts were stable over time, and showed no change in detector response when stored at 0°C for 20 and 2 weeks, respectively. Soil samples containing RE-40885 residues were stable for 12 months when stored at −15°C.

Recoveries for soil 1 ranged from 77 to 110% of applied

Received August 7, 1989 Accepted October 16, 1989.

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Table 1. Soil characteristics

Soil	Series	Sand, %	Silt, %	Clay, %	Organic matter,[a] %	pH[b]
1	Greenville	51	24	25	2.1	6.4
2	Cecil	50	37	13	1.4	5.2
3	Dothan	83	11	6	0.5	7.0

[a] Determined by Walkley-Black dichromate method.
[b] Determined by 1:1 soil:water suspension technique.

Table 2. Solvent systems for RE-40885 analysis

Mobile phase	Solvent ratio, v/v	Retention time, min	k'[a]
Acetonitrile–water	80 + 20	3.30	0.65
Acetonitrile–water	60 + 40	6.99	2.50
Methanol–water	80 + 20	5.11	1.55
Methanol–water	72 + 28	7.00	2.50
Methanol–water	70 + 30	9.42	3.71
Methanol–water	60 + 40	22.60	10.30
Acetonitrile–2-propanol	80 + 20	2.82	0.41
Acetonitrile–2-propanol	60 + 40	2.73	0.36

[a] Capacity factor; t_0 for system = 2.00 min.

Table 3. Determination of RE-40885 recovery from soil 1 with methanol extraction, methanol–water (72 + 28) mobile phase.

Spiked concn, ng/g	Detected concn,[a] ng/g	RSD, %
0	0	0
40	43.0	23.1
50	41.4	24.1
80	76.5	12.0
100	97.6	1.1
120	107.0	3.7
160	155.8	5.1
200	220.7	3.9
250	193.0	6.7
500	451.0	4.6
1000	927.0	4.0

[a] Mean values obtained from 3 flasks. Average recovery = 94%.

herbicide, with the average recovery being 94% (Table 3). The precision of the analytical procedure over the range of 40–1000 ppb was acceptable, with the average relative standard deviation over this range being 8.0%. This analytical range corresponds to soil concentrations of approximately 400–600 from a RE-40885 field application. Our limit of detection for quantitation was defined as 40 ppb; where the signal-to-noise ratio was 3 (Figure 1c). However, qualitatively, the limit of detection of RE-40885 may be extended as low as 20 ppb. Results for soils 2 and 3 were similar to soil 1.

Herbicides, including RE-40885, often are applied in combination with other pesticides. The pesticides examined for detection interference represent major classes of pesticides used in crops where RE-40885 has potential for use. These commonly used chemicals did not elute in the retention window of RE-40885, and did not interfere with the method. Pesticides that eluted and their retention times in minutes were: atrazine (6.25), chlorothalonil (10.05), alachlor (17.5), and trifluralin (18.28). Benefin did not cause a detector response with this method.

Acknowledgments

The authors thank Rick Arrendale for obtaining and interpreting the GC/MS spectra.

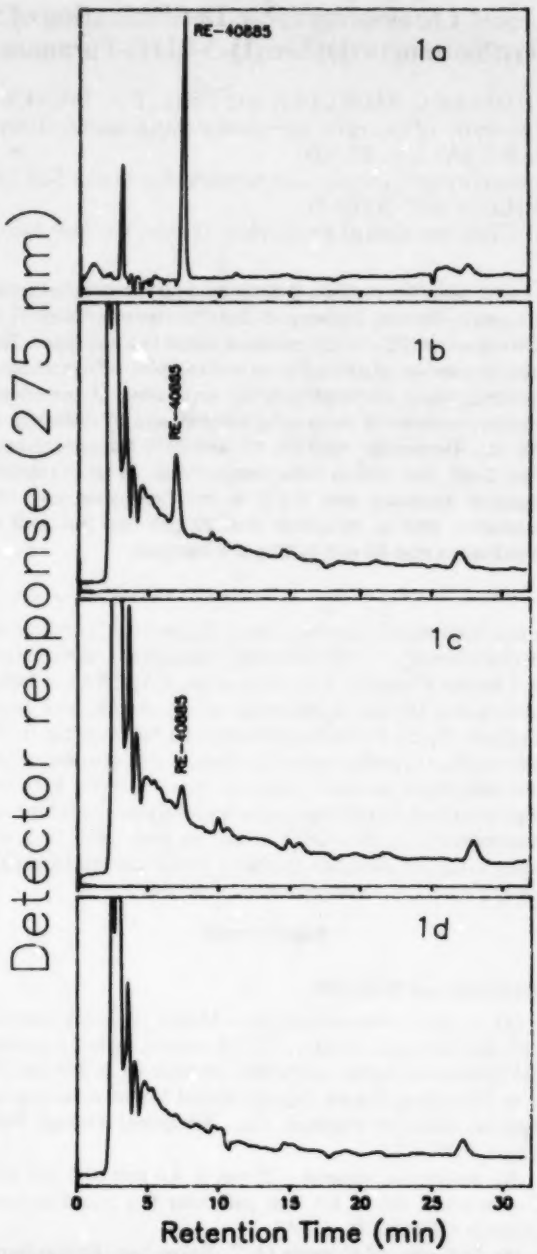

Figure 1. Liquid chromatograms of analytical standard and methanol extracts of soil spiked to achieve various RE-40885 concentrations: a, 200 ppb RE-40885 analytical standard; b, Soil 1 extract containing 120 ppb RE-40885; c, Soil 1 extract containing 40 ppb RE-40885; d, Soil 1 extract containing no RE-40885.

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